available. They operated at a superficial face velocity of 3 cm/sec, an initial pressure drop of 2.0 kPa, and an activity reduction of 99.7 percent. Additional units were built at the Savannah River Site (SRS) later, and each has given many years of continuous service. It should be noted that the design airflow resistance of deep-bed sand (DBS) filters is higher and the retention efficiency lower than may be obtained using absolute filters, but freedom from servicing and replacement over many years are important advantages when the collected material is intensely radioactive. In addition, DBS filters are nonflammable, largely unaffected by condensed water and strong acids, and provide a substantial heat sink in the event of fire or explosion. They are, however, also large, expensive, and nondisposable.

Rapidly emerging glass fiber technology during the 1940s and 1950s shifted attention to the use of very deep beds (250 cm or more) of curly glass fibers, combined with HEPA-quality final filters, as a satisfactory substitute for sand filters when treating gaseous effluents from chemical operations.[73] These proved to be more efficient and to have lower airflow resistance than the sand filters they replaced. Deep-bed glass fiber filters have been used at Hanford for several decades on their Purex process effluent stream, and a similar installation is in place at the DOE Idaho chemical plant. In addition, an investigation was conducted to determine the ability of a similar type of deep-bed fiber filter to collect and store, at modest pressure rise, very high loadings of sodium-potassium fume that might accidentally be released from a liquid metal fast breeder reactor.[19]

There has been interest in sand filters for emergency containment venting for light-water reactors. An installed Swedish containment venting system known as FILTRA features large concrete silos filled with crushed rock. These silos were designed to condense and filter steam blown from the containment and to retain at least 99.9 percent of the core inventory.[23] Later designs for containment venting utilized wet systems to remove gaseous radioiodine.[23]

1.3 BRIEF HISTORY OF GAS ADSORPTION

1.3.1 INTRODUCTION

Iodine in its many chemical forms is probably among the most extensively studied fission products produced in the nuclear industry. The generation, release mechanism, properties, forms, trapping and retention behavior, and health effects of iodine-131 have been the subject of numerous studies, but a comprehensive understanding of the significance of its release to the environment and integration of the chemical technology into protection technology may remain incomplete in some aspects. The technology associated with the removal and retention of all iodine isotopes is similar to that for iodine-131, but interest in removal efficiency has shifted somewhat toward the importance of long-term retention with the increasing half-life of the iodine isotope.

A removal technology for the radioactive noble gases (krypton, xenon, radon) using adsorbents has also been studied extensively. This removal technology has become a standard control method for boiling water reactor (BWR) offgas decontamination and has replaced pressurized tank retention for pressurized water reactor (PWR) offgas control. A similar technology can be used to hold up the krypton-85 contained in reprocessing offgases.

Volatile metal compounds such as ruthenium and technetium can be removed from gas streams by adsorption, but a solid-surface-supported chemical reaction is often necessary for good retention. Removal technologies for carbon-14 and tritium also involve the use of adsorbents, either as collecting agents or as catalysts for conversion to other, more easily removed compounds.

Vapor recovery by adsorption was a well-established chemical engineering unit operation process prior to nuclear technology development for weapons and power production. Generally, vapor recovery systems utilized beds of activated carbon that were 60 cm deep or more and often consisted of two or more identical units in parallel so that one could be on-stream while a second was being desorbed by low-pressure steam and a possible third was undergoing cooling after steam desorption. These multi-bed arrangements made

continuous operation of vapor production processes possible.

Adsorbents of various types, both impregnated and unimpregnated, became widely used during and following WWI in military and civilian gas mask canisters and cassettes for removing a wide range of toxic substances from breathing air. Activated carbon derived from nut shells was used in the U.S. Army service gas mask during WWI. Later, the activated carbon used in the service gas mask was derived from coal and impregnated with metals that catalyze reactions with gas warfare agents. Activated carbon also was used to treat ventilation air in special applications such as removing sulfur dioxide and ozone from air supplied to libraries housing rare book collections to prevent paper embrittlement. Ventilation applications used shallow beds of activated carbon, generally 2.5 cm or less, because complete removal of outdoor contaminants was seldom a requirement and low airflow resistance was essential to prevent unacceptable fan noise levels. The theoretical basis for adsorption processes was greatly advanced by the need to develop gas mask applications during WWI, and an early theoretical analysis of physical adsorption was made by Langmuir.[22] Thus, there was a considerable body of knowledge available on the application of adsorbents, especially for activated carbon, when the nuclear industry developed a need for this technology.

Control of iodine emissions from chemical processing of spent nuclear fuel was initially done by liquid scrubbing using caustic solutions, and sometimes with the addition of sulfate salts, but retention efficiency by scrubbing seldom exceeded 90 percent. To improve iodine retention efficiency for dissolver offgas cleaning, activated carbon beds were added to the caustic scrubber at the DOE's Idaho Falls Plant in 1958 and were reported to provide additional decontamination factors of 10 to 30. Silver-plated Fiberfrax fibers also were investigated at the Idaho Test Station for use as a combined particulate filter and iodine retention device for hot calciner offgas cleaning. Other studies of this nature were conducted with silver-plated copper filaments, and an iodine decontamination factor of 10 was reported.

Iodine releases to the atmosphere in the event of a reactor accident became a major concern as the nuclear industry began its rapid expansion during the early 1960s, and attention focused on iodine removal during normal and abnormal conditions at ambient and elevated temperatures. An iodine decontamination factor of 10 was reported. At ORNL, studies were conducted on activated carbon beds for the holdup of radioactive fission gases generated during the operation of nuclear reactors and during nuclear fuel reprocessing. The principal area of interest was delaying release until short-half-life isotopes decayed to levels that were acceptable for release. This approach utilized conventional theoretical plate equations.

1.3.2 POWER REACTORS

The first major U.S. effort related to control of radioiodine from reactors consisted of design studies of confinement systems for the nuclear-powered commercial ship N.S. Savannah and the Hanford N Reactor.[32, 33, 34] At that time, control of elemental iodine was of primary interest, mainly because data from various prior accidents failed to differentiate iodine forms. An early and very realistic analysis and solution of iodine retention requirements was presented by Riley from the United Kingdom. He proposed a process-engineering solution to iodine retention and recommended 30-cm-deep carbon beds operated at high velocity and with 0.5-sec residence time. In the United States, however, the heating, ventilating, and air-conditioning (HVAC) shallow-bed model was adopted by the nuclear industry, and shallow beds of carbon became the predominant method for iodine capture.

The U.S. activated carbon adsorber design was based on a series of relatively short-term laboratory experiments using fresh carbon, clean carrier gas, and nonsystematic iodine inlet concentrations.[35, 36, 37, 38] Results indicated an iodine removal efficiency for 2.0- to 2.5-cm-deep carbon beds that could not be obtained in practice. Typically, the early installations were constructed in pleated form and contained 20 to 25 kg of carbon for every 5 m³/sec of airflow.[39, 40, 41] This design became known as a Type I Adsorber Unit. It was later found that, under high-humidity conditions (greater than 70 percent relative humidity), shallow carbon beds were incapable of high-efficiency removal of organic iodides, particularly methyl iodide.[42, 43, 44] The

somewhat accidental discovery that isotope exchange would take place on carbon surfaces and that gas mask carbons impregnated with tertiary amines to control low-molecular-weight chemical warfare agents containing organic halides would also react with radioactive organic halides led to the use of carbons impregnated with stable iodine or iodide salts to control methyl iodide by isotope exchange, as well as the use of amine-impregnated carbons to control methyl iodide by complex formation.[45, 47, 48, 49]

Although laboratory experiments with unimpregnated carbons indicated that a 2.5-cm bed performed acceptably for elemental iodine removal when the exposure was a short duration and the carbon was fresh, a minimum acceptable bed depth of 5 cm was needed under ideal conditions for the impregnated carbons used for methyl iodide removal. This led to development of a tray-type design for nuclear adsorber units consisting of two 5-cm-deep military-type[50] adsorber trays that were attached to a 61- x 61-cm face plate for mounting in ladder frames. This adsorber design became known as a Type II Adsorber Unit. It provided a 0.25-sec gas residence time in the carbon and operated at a gas velocity of 20 cm/sec.

Standardization of the external dimensions of the tray-type units did not occur for many years, and there are currently approximately ten different adsorber sizes in service in the United States. This creates logistical difficulties for warehousing spares and obtaining fast replacements in case of an accident. For example, between the two reactors on the TMI site at the time of the TMI-2 accident, there were four different adsorber shapes and sizes—three of them supplied by the same vendor.

In the beginning, criteria for the selection of adsorbent media were not well standardized in the United States. Based on short-term tests, carbon impregnated with potassium iodide and iodine performed better than unimpregnated carbon and its use dominated early iodine control technology. A water extract from finished impregnated carbons varied in pH from neutral to acidic depending on the method of preparation. As the pH of the water extract of the base carbon also influences the pH of the impregnated carbon, the choice of vegetable-base (coconut shell) carbons

for impregnation was helpful because, in addition to being hard, such carbons contain approximately 1 percent potassium hydroxide or sodium hydroxide that reacts with free elemental iodine to produce iodide forms that migrate through the carbon less easily than elemental iodine.[51, 52, 53]

In the late 1960s and early 1970s, researchers realized that design data derived from short-term experiments with fresh carbons provided inadequate adsorber designs for the long-term protection needed from carbon beds. Carbons deteriorate from long exposure to air pollutants (weathering), as well as from inadvertent adsorption of widely used organic compound-containing materials (poisoning). Both situations result in a loss of capacity for iodine species. Such observations led to development of deep-bed adsorbers constructed with 10- to 50-cm-deep beds of impregnated carbon that could be filled by pouring the granules into large panels, thereby eliminating the many leak paths associated with tray-type units.[54, 55, 56, 57] This adsorber design was designated a Type III Adsorber.

The nuclear reactor post-accident iodine release concepts that became established and codified during the late 1960s were based on the assumption that a large quantity of elemental iodine would be released and have to be adsorbed. The design criteria were based on the release of 50 percent of core iodine with half of the released iodine captured by plate-out on surfaces. Of the remaining airborne iodine, 85 percent would be elemental, 10 percent would be organic, and 5 percent would be particulate. Contemporary transport concepts contemplated a need to treat large air volumes at locations several steps away from the point of release of the iodine fission products. It was anticipated that iodine capture would be made more difficult by dilution in a large volume of air, as well as by the presence of a large quantity of other chemicals in the air that would compete with iodine for adsorption sites or react more rapidly with the impregnants.

1.3.3 RADIOCHEMICAL PROCESSING

The quantity of radioiodine used in radioactive tracer studies is small compared to the concentrations present in power reactors, but the variety of radioiodine-containing organic compounds is greater. Based on available

theoretical and experimental data, the removal efficiency of impregnated nuclear carbons for many organic compounds is lower than for methyl iodide. Furthermore, most radioiodine decontamination systems found in connection with laboratory fume hoods are inadequate even for methyl iodide, as they usually only contain a depth of 2.5 cm of some unimpregnated carbon that has not been specifically qualified for this intended use. For laboratory hood service, carbon depth should provide at least a 0.25- to 0.50-sec residence time and should permit removal of representative samples for periodic laboratory testing to determine remaining service life. Representative samples should be removed at least every 720 hrs of continuous use and should be tested under conditions corresponding to the hood effluent conditions with respect to relative humidity, temperature, and the presence of compounds that compete with the radioiodine species for adsorption sites. For example, when relative humidity is variable, the adsorbent should be tested at the maximum relative humidity conditions likely to be present to obtain conservative values.

1.3.4 FUEL REPROCESSING PLANTS

The isotope of importance in the effluent gases from fuel reprocessing systems is iodine-129, which has a half-life of 1.7 x 10[7] years. Generation of gaseous iodine-129 occurs in the presence of oxidizing acid gases such as nitrogen oxide under very-high-humidity conditions, and often when there are high concentrations of competing organic compounds. This is a very demanding environment for adsorption media. At the beginning, reprocessing effluent treatment in the United States usually involved liquid scrubbing with alkaline solutions. However, there are anecdotal reports of a packed bed scrubber at Hanford that utilized silver dollars for the packing to make the captured iodine more insoluble as silver iodide. Although alkali, mercuric nitrate, and hyperazeotropic nitric acid absorption systems are still used for this purpose, direct removal of iodine using solid adsorbents has been gaining favor in treating the gaseous effluent at newer fuel reprocessing plants. The use of solid adsorbents for this service was first evaluated at the SRS with activated carbon, but it proved to be unstable in the dissolver offgas environment. In 1968, a

switch was made to a silver-impregnated inorganic adsorbent. The solid adsorbents under consideration include primary silver-containing materials such as silver-exchange zeolites and silver-impregnated adsorbents. In this case, the adsorbent acts as a carrier for the silver-iodine chemical reaction. Due to the relatively high cost of silver, it is important that as much silver as possible is utilized before exhaustion of the adsorbent system. Numerous studies have been conducted to evaluate these materials for full reprocessing service.[58, 59, 60, 61]

The most commonly used adsorbents for dissolver offgas treatment include AC6 120, a silver-nitrate-Impregnated, high-silica-base adsorbent;[62] a silver-and-lead-nitrate-impregnated, high-temperature base adsorbent;[63] and silver-exchange zeolites[64] and mordenites.[65] Several reaction mechanisms lead to various silver-iodine compounds. The most common compound for both elemental and organic iodine is silver iodide, which is very stable except in a high-temperature hydrogen environment where reduction to elemental forms occurs.

1.3.5 USE OF ADSORBENTS FOR NOBLE GAS CONTROL

The application of adsorbents for noble gas retention was developed at ORNL. The concept involves self-regeneration of the adsorbent due to decay of the noble gases to solid daughter products as they pass through very deep adsorbent beds that require a long time for passage and result in the successive extinction of noble gas radioisotopes—those with the shortest half-lives disappear first. This technology is generally used to decontaminate all noble gas isotopes (except krypton-85 because of its long half-life—nearly 11 years). The process is particularly well suited to treat BWR off-gas streams and was applied first at the KRB site in Germany. The first BWR installation in the United States was the Interim Offgas System at the Vermont Yankee Plant.[66] It was succeeded by the Advanced Offgas System at the same site.[67] The earlier technology involved ambient temperature systems. Cooled or refrigerated systems were later designed by General Electric Company.[65]

Storage tanks were used for PWR degasifier gas processing at first, but a continuous-flow

adsorption system was installed at Seabrook Nuclear Power Station, the first for a U.S. PWR.[69] Design parameters for noble gas adsorption systems were established on a more systematic basis than was the case for control of radioiodine, and the few problems that have occurred with these plants were related to improper humidity control or accidental wetting of the carbon prior to operation. Two temperature excursions have been reported in these systems—one at ORNL, where an oxygen stream was being decontaminated,[70] and one at Browns Ferry nuclear power plant, where a hydrogen recombiner malfunctioned.[2]

1.4 ADSORBENT BED SIZING FOR NOBLE GAS DELAY

The sizing of adsorbers for delay of radioactive gases has been evaluated by several investigators.[1] Browning developed a theoretical chamber model which, although not a precise description of the dynamic adsorption process, has been empirically found to follow the performance of delay beds. The application of the theoretical plate concept for krypton-xenon delay bed calculations was made by Burnette.[71] This process assumes nonequilibrium conditions where molecular diffusion through the stagnant gas film in contact with the carbon surface is the rate-limiting step. Bruck[91] devised a very simple concept based on a proportionality factor to describe the distribution of the adsorbate between the adsorbent and the interstitial space between the adsorbent grains. Close examination indicates that the term "dynamic adsorption coefficient" used in the design of these systems is also the slope of the Langmuir adsorption isotherm when equilibrium conditions exist. The adsorption coefficient can be determined in both under static (K') and dynamic (K) conditions. The static coefficient represents equilibrium conditions between the gas phase and the adsorbed component of the adsorbate, whereas the dynamic coefficient represents the sum of the equilibrated and the nonequilibrated (caused by mass transfer limitations) portions of the bed. The ratio of $K/K' \approx 1.0$ applies to very large systems that contain the same adsorbent when temperature, pressure, and adsorbate and co-adsorbate concentrations are the same.

Testing of Iodine Adsorbents

The current test protocol is ASTM D3803-79, which superseded RDT M-16. Both standards have numerous typographical and editorial mistakes such as inaccurate decay constants for iodine-131 and inconsistencies in time duration between the text and tables. Both procedures are merely guides as far as equipment setup is concerned, but the critical parameters listed in Table No. 1 of ASTM D3803-89 and Section 13 specify reporting requirements.

Testing of Noble Gas Adsorbents

The results of noble gas delay are not correlatable because important test parameters either were not reported or were not standardized. Omissions include the unspecified moisture content of the adsorbent, relative humidity of the gas, and duration of pre-equilibration for the experiment. In some cases, tests involved only a few grains of carbon, and the results have been extrapolated to full-size systems with bad results.

Operating Experience with Iodine Adsorption

Several important lessons concerning iodine control were learned from the TMI-2 accident. The first is that the conventional iodine release and transport theories were incorrect. Most of the iodine stayed in the liquid phase or plated out in the containment vessel. The total amount of iodine that reached the operating filter adsorber trains can be conservatively estimated at 150 Ci, of which approximately 15 to 32 Ci were released to the environment. This value, when compared with approximately 13×10^6 Ci of xenon-133 released, is a good indication of the lack of predicted partitioning of iodine species into the airstream. One indication of the iodine species distribution showed a predominance of methyl iodide, followed by elemental iodine. The system available for controlling iodine releases was comprised of two trains in the Unit 2 Auxiliary Building, identified as trains A and B, and two trains in the Fuel Handling Building, identified as trains A and B. The Auxiliary Building trains were not classified as engineered safeguard facilities. They captured approximately 12 to 14.6 Ci of iodine and released approximately 1.2 to 1.8 Ci. The Fuel Handling Building filters were downgraded safeguards that captured

approximately 36 to 48 Ci of iodine and released approximately 5 to 15 Ci.

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